

## Interpretation of Low-Temperature Data Part VI: The Magnetite Verwey Transition (Part C): Low-Temperature Demagnetization of Stoichiometric Magnetite

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*In this installment of the ongoing IRM Quarterly series on low-temperature magnetism, we return for a third time to the Verwey transition in stoichiometric magnetite. The first magnetite article (IRM Quarterly, v. 20, n. 4, 2011) provided an overview and focused on the physical nature of the transition. The second article (IRM Quarterly, v. 21, n. 4, 2011) discussed the effects on remanence, susceptibility, and hysteresis of cooling in an applied field across the Verwey transition, in both the single domain and multi-domain case. The so-called field-cooled (FC) / zero-field-cooled (ZFC) experiment is a common one performed at the IRM, and we discussed the interpretation of FC/ZFC data. In this article we address the Verwey transition in the context of another common experiment performed at the IRM: low-temperature demagnetization of a remanence acquired at room-temperature. We again restrict our discussion to stoichiometric magnetite, leaving cation substitution, oxidation, stress, and pressure effects for future articles in this seemingly inexhaustible series.*

### Introduction

Low-temperature demagnetization (LTD) of magnetite (by cycling a sample from room temperature to a temperature  $T < T_V \sim 122\text{K}$  and back, in zero field) has a long and distinguished history (e.g., Ozima et al., 1964; Kobayashi & Fuller, 1968; Merrill, 1970). Despite many years of study, however, LTD still retains some significant mysteries. In contrast to the warming of low-temperature remanences (IRMQ v. 21 n. 4), where properties change abruptly and dramatically when the strong monoclinic magnetocrystalline anisotropy vanishes at  $T_V$ , the changes on cooling from room temperature can be more gradual, subtle and complex: the weak cubic magnetocrystalline anisotropy diminishes progressively until the easy axis orientation changes at the isotropic point  $T_K \sim 130\text{K}$  (from

body diagonal to cube edge), and finally the monoclinic phase nucleates at  $T_V$  and propagates through the individual particles. Moreover, the initial state for LTD experiments is often subtle and complex as well (natural remanence, or weak-field lab analogues), whereas FC/ZFC experiments typically involve brute-force strong-field remanences. For these reasons, the behavior we observe during zero-field cooling and rewarming can exhibit a rich variety of features.

For many years there were practical hurdles to measuring magnetization during complete cooling/warming cycles. Commonly the effects of low-T cycling were studied solely through measurement of remanence at room temperature, before and after treatment by immersion in liquid nitrogen in a zero-field environment (e.g., Merrill, 1970). Some of the key early observations of magnetization during cooling/warming cycles (Yama-ai et al., 1963; Nagata et al., 1964; Ozima et al., 1964; Kobayashi & Fuller, 1968) involved the newly-developed vibrating-sample magnetometer (Flanders and Doyle, 1962). Later work involved continuous measurements on specialized instruments including a Dewar-equipped spinner magnetometer (Hartstra, 1983) and a SQUID susceptometer (Halgedahl & Jarrard, 1995). At the IRM, however, the workhorse Quantum Designs MPMS instruments were unable to measure during cooling, due to hardware and software limitations, until about 1998 (IRMQ v. 8, n. 4; Özdemir & Dunlop, 1999). Prior to that all low-T experiments at the IRM were based solely on measurements during warming, and it has only been over the last decade or so that we have accumulated a large body of data on the behavior of magnetite-bearing materials during complete low-T cycles (e.g., Fig 1).

### Pioneering Studies

The earliest studies of changes in room-temperature remanence during low-T cycles were designed to understand (a) the fundamental origins of stable remanence in multidomain grains and the relative importance of stress, microstructures, magnetocrystalline anisotropy and particle geometry, and (b) the nature of the low-temperature

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# Visiting Fellow's Reports

## How does magnetization vary in the lower crust? Rock magnetic studies from the Athabasca Granulite Terrain, Canada

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Introduction: SWARM, a trio of satellites observing the Earth's magnetic field with a launch date of 2011, will provide detailed information on the magnetization of the Earth's crust and the long wavelength magnetic anomalies (LWMA) produced there. Although not the only mission of SWARM, mapping of lithospheric magnetization and the resulting geologic interpretation is an important part of this immense project. The last large collection of satellite magnetic data, the MAGSAT project in the early 1980s, provided information on magnetic anomalies of crustal source of some +/- 15 nT. Interpretation of these anomalies required 2 to 5 A/m of magnetization in lower crustal rocks, but studies did not find sufficient susceptibility or magnetization in the lower crust (Shive et al., 1992). Much is known about the magnetic properties of the crust (Purucker and Whaler, 2007), but we still do

not fully understand what is magnetic in the lower crust and what is the source of the LWMA. Early studies only considered susceptibility and induced magnetization; remanence was not considered as a possible contributor.

The Athabasca Granulite Terrane, between the Proterozoic Rae and Hearne Blocks of the Churchill Province of north-central Canada is an excellent region to study the rock magnetic properties of lower crustal rocks. It is the largest exposure of lower crustal rocks on the Earth's surface, composed of suites of both felsic and mafic granulite facies rocks metamorphosed at ca 1.0 GPa pressure. They have resided in the deep crust at depths of ~35 km from ~2.6 Ga to ~1.8 Ga when they were exhumed (Williams and Hanmer, 2006). Exposed rocks include tonalite, granite, felsic granulite, mafic granulite, and mafic dikes. Extensive geologic information is available from Mike Williams (UMass), colleagues and students (e.g. Dumond et al., 2008; Flowers et al., 2008). Aeromagnetic coverage of this region is excellent, and the resulting anomaly map is quite exquisite (Fig. 1), showing some direct relationships between rock type and anomalies and other examples where anomalies cut across mapped units.

What I did at the IRM: A total of forty-two samples representing all major rock types in the area, and coming from both magnetic high and low regions were brought to the IRM to augment initial susceptibility and NRM measurements. Room temperature hysteresis on all samples revealed a wide range of properties, with  $M_r/M_s$  ranging from 0.01 to 0.9 and  $H_{cr}/H_c$  ranging from 1.2 to 13.4. Samples from the negative anomaly areas generally plot with  $H_{cr}/H_c$  less than 4.0 indicating PSD

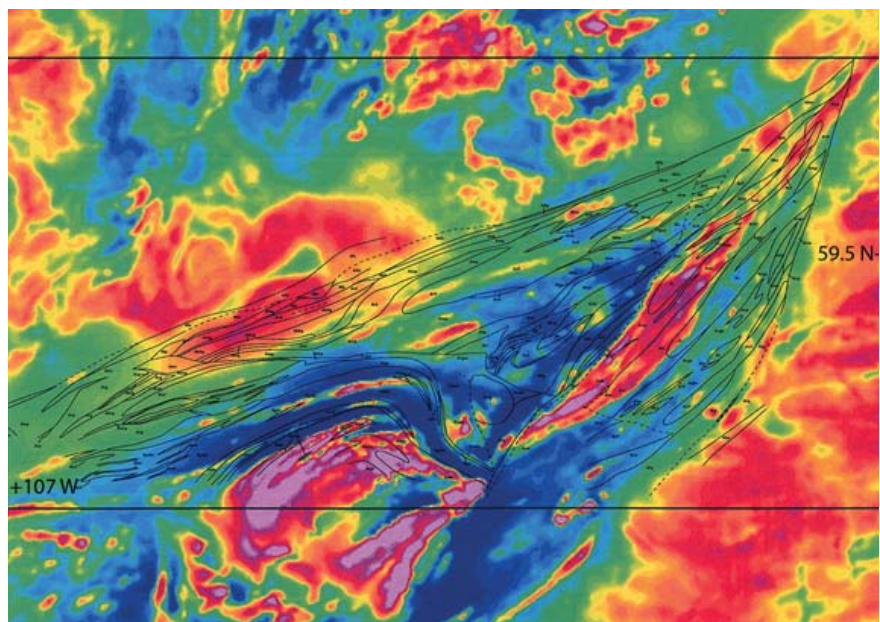


Figure 1. Aeromagnetic anomaly map of a section of the Athabasca granulite terrain, northern Saskatchewan, Canada, with geologic contacts overlain. Data from the Geological Survey of Canada, map processed by C. Koteas, UMass. Total anomaly range  $\approx 2000$  nT. Width of map area  $\approx 100$  km. (see full-color version at <http://www.irm.umn.edu/quarterly/irmq22-2.pdf>).

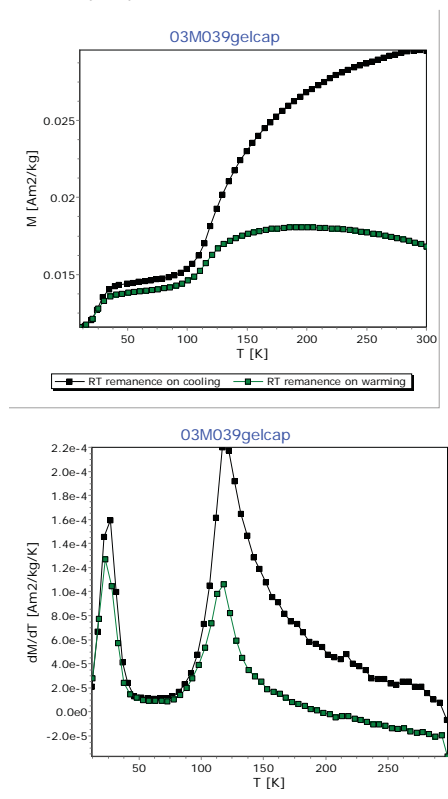


Figure 2. RTSIRM data on sample 03M039 (diatexite from magnetic high region), showing presence of both magnetite and pyrrhotite. Upper plot - magnetization recorded with temperature; lower plot - first derivative of the above data.

to SD behavior, while most of the samples from the positive anomalies areas plot in the MD range. It should be noted that for a number of samples the actual values of  $M_r$  and  $M_s$  were very low and loops were not always well defined, indicating the dominance of paramagnetic behavior in these samples. The majority of well-defined loops indicated magnetite as the primary oxide. Despite extensive investigation, no samples have yet produced “fat” loops indicative of hematite or hemo-ilmenite as the predominant oxide mineral.

Investigations of sample behavior at low temperatures using the MPMS system provided interesting although sometimes confusing results. A number of samples, especially those from the tonalite, showed clean and distinct Verwey transitions, as expected for magnetite-dominated rocks; in some samples pyrrhotite is also evident with a drop in magnetization around 30-35°K (Fig. 2). Additional samples produced a Verwey transition, but one of a broad nature extending well over 50° and often with two distinct peaks. A few samples showed only pyrrhotite signatures on heating and cooling. And, sadly enough, a number of samples had little or no indication of diagnostic magnetic behavior during the MPMS runs, indicative of the paramagnetic nature of some of these samples. This behavior was most noticeable in the dike samples, already known to have low susceptibilities ( $<10^{-3}$  SI) and weak magnetizations.

The simplest conclusion of this experimental work at IRM is that the rocks in the Athabasca Granulite Terrain include samples with well-behaved magnetite but also samples with little or no magnetite, leading to the observation that the extreme magnetic anomalies are related to large swatches of juxtaposed “magnetic” and “non-magnetic” lower crustal rocks, even though many of them have similar rock descriptions. Work is continuing to investigate specific rock compositions, magnetic properties, and geologic histories to better understand what is magnetic in this part of the lower crust.

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# Visiting Fellows

## July - December, 2012

### Trevor Almeida

Imperial College London

*Characterisation of glass-ceramic samples of magnetite and titanomagnetite*

### Mitch Campion\*

Concordia College

*Magnetic investigation of e-Fe2O3 formed after maghemite thermal treatment*

### Stephen Garrett\*

Texas State University-San Marcos

*Geomagnetic Study of Burned Rocks at the Gault Site (41BL323), Central Texas*

### Sara Gillooly

University of Pittsburgh

*Validating tree leaf use as a novel method to reduce exposure misclassification*

### Plinio Jaqueto

University of São Paulo

*Earth's magnetic field variations in the past millennia from Brazilian speleothems*

### Mathilde Meijers

University of Nice-Sophia Antipolis

*Characterizing rock magnetic behavior of remagnetized Paleozoic limestones from the South Armenian Block (Lesser Caucasus) and the central Taurides (southern Turkey)*

### Gary Stinchcomb

Baylor University

*Climatic, geomorphic and human controls on the magnetic properties of soil along the middle Delaware River valley, USA*

\*U.S. Student Fellows

The next Visiting Fellowship application deadline is October 30.



*Oncorhynchus mykiss*



*Ficus benjamina*



*Krafla lava ropes*

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## Current Articles

A list of current research articles dealing with various topics in the physics and chemistry of magnetism is a regular feature of the IRM Quarterly. Articles published in familiar geology and geophysics journals are included; special emphasis is given to current articles from physics, chemistry, and materials-science journals. Most abstracts are taken from INSPEC (© Institution of Electrical Engineers), Geophysical Abstracts in Press (© American Geophysical Union), and The Earth and Planetary Express (© Elsevier Science Publishers, B.V.), after which they are subjected to Procrustean culling for this newsletter. An extensive reference list of articles (primarily about rock magnetism, the physics and chemistry of magnetism, and some paleomagnetism) is continually updated at the IRM. This list, with more than 10,000 references, is available free of charge. Your contributions both to the list and to the Current Articles section of the IRM Quarterly are always welcome.

## Anisotropy and Magnetic Fabrics

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## Tectonics/Paleomagnetism

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Fig 1. Anatomy of a low-T cycle of RTSIRM (initial magnetization M1). Black squares show cooling curve reaching a minimum (M2) at or near  $T_v$ , followed by an increase to a local maximum M3 and subsequent decline to M4 at the minimum temperature (in this case 10 K). On rewarming (green diamonds) the path retraces the cooling curve through M3 and M2 and reaches a slightly lower minimum (M5) before recovering somewhat on returning to M6 at room temperature. (unpublished data from P Kelso, granite from the Idaho Batholith)

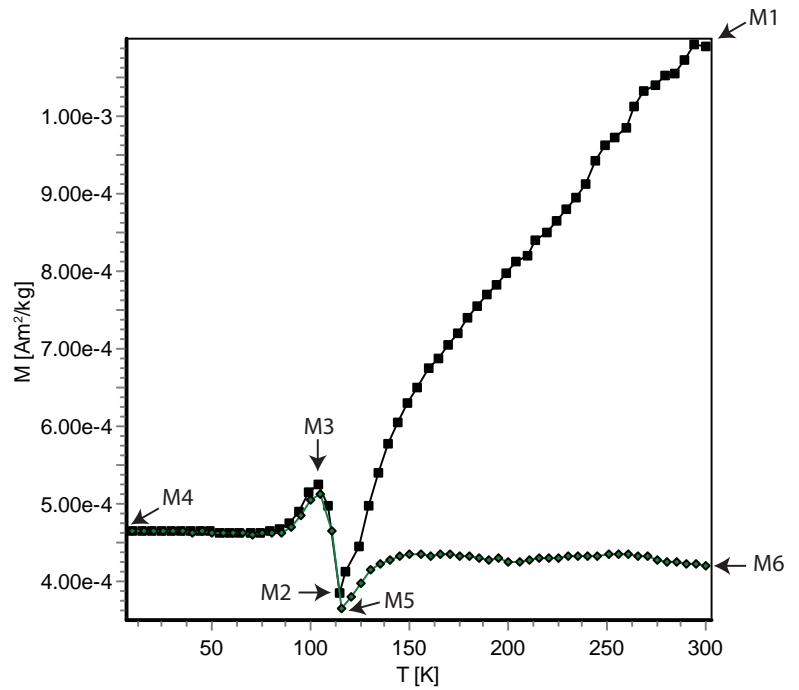
Note that not all of these features are generally seen in RTSIRM low-T cycles; for many/most natural magnetites,  $M5 \approx M4 \approx M3 \approx M2$ .

Many parameters have been introduced to describe behavior during such experiments. The simplest is often called the “memory ratio,” equal to  $M6/M1$ , i.e., the proportion of initial remanence that survives the low-T cycle (e.g., Dunlop & Argyle, 1991; Muxworthy & Williams, 2006). This can be determined without measurements at low T, and is therefore readily quantified. The same quantity is called ‘second memory’ by Özdemir et al (2002), to distinguish it from ‘first memory’ ( $=M4/M1$ ), the proportion of RTSIRM that survives the first crossing of the transition. King & Williams (2000) define “memory” as the magnetization recovered on rewarming through the transition, i.e.,  $M6-M5$ . For cases where there is a distinct jump in remanence on crossing the transition, the parameter  $\Delta_{vj}$  (Muxworthy et al, 2003) is defined as size of the discontinuity, normalized to the initial remanence, i.e.,  $\Delta_{vj} = (M3-M2)/M1$ ; it can have positive values (as in the figure) or negative values. Other parameters known as  $\Delta M_m$  and  $\Delta M_c$  (Özdemir & Dunlop 2010) respectively denote magnetization differences in the monoclinic and cubic phases:  $\Delta M_m = M4-M5$ , and  $\Delta M_c = M1-M6$ .

transitional phenomena themselves. In particular, for many years there was no clear evidence to show the relative importance of the magnetocrystalline anisotropy transition and the Verwey transition in low-temperature demagnetization (Halgedahl & Jarrard 1995; Özdemir & Dunlop, 1999; Dunlop, 2003, Liu & Yu, 2004).

Three notable properties of magnetite’s LTD behavior were noted almost immediately (e.g., Ozima et al., 1964; Kobayashi & Fuller, 1968; Merrill, 1970): (1) multidomain grains are demagnetized much more effectively than single-domain grains; (2) strong-field remanence is erased to a much greater extent than weak-field remanence (such as TRM); and (3) multiple low-T cycles generally demagnetize more completely than a single cycle. Kobayashi & Fuller (1968) speculated that the origin of “memory” (the remanence that survives LTD) is related to the origin of TRM.

Ozima et al (1964) showed experimentally that soft (1-10 mT) isothermal remanent magnetizations (IRMs), superposed on TRM or NRM, could be preferentially re-



moved by low-temperature zero-field treatments. Yama-ai\* et al (1963) observed a phenomenon that we have never seen reproduced, namely a reversal of magnetization in a single crystal on cooling through 130 K, followed by a return to the original polarity on rewarming (fig 2), and they hypothesized a mechanism involving two sources of magnetization, weakly coupled magnetostatically, since the self-reversal could be suppressed by application of a weak field during cooling. Kobayashi & Fuller (1968) carried out several experiments to test this idea further, including (a) comparison of room-T AF demagnetization spectra of RTSIRM and of “memory” after LTD, showing that the latter has much higher stability, and (b) superposition onto a room-T SIRM of a soft reverse-polarity low-T IRM, showing that “memory” is independent of the soft low-T overprint. They argued that a magnetostatic model could not explain these observations and that internal stresses must play a key role.

## Effects of Domain State

### Single Domain, low-T cycling of RTSIRM

In an SD particle at  $T > T_v$ , magnetic remanence lies along a minimum-energy orientation between one of the cubic  $\langle 111 \rangle$  easy axes and the axis of particle elongation; the precise orientation depends on the relative strengths of the magnetocrystalline anisotropy and the shape anisotropy, the latter usually being stronger. On cooling through the isotropic point at  $T_k = 130K$ , the cube edge orientations  $\langle 100 \rangle$  become the magnetocrystalline easy axes, and then with the crystallographic change at  $T_v = 120K$ , the monoclinic c-axis  $[001]$  becomes the strongly-preferred

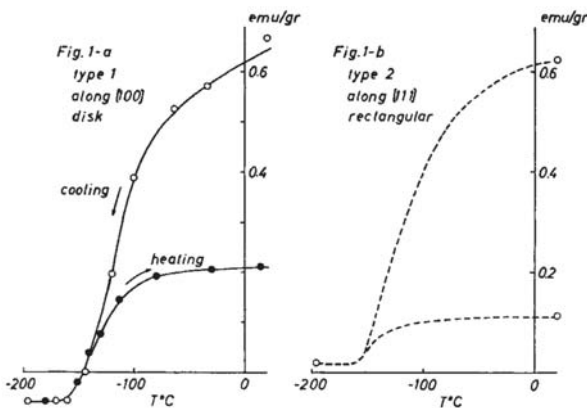


Fig 2. Yama-ai et al, Nature 1963; note self reversal along  $[100]$ .

\*Mituko Yama-ai married Minoru Ozima and her later papers are written as Mituko Ozima

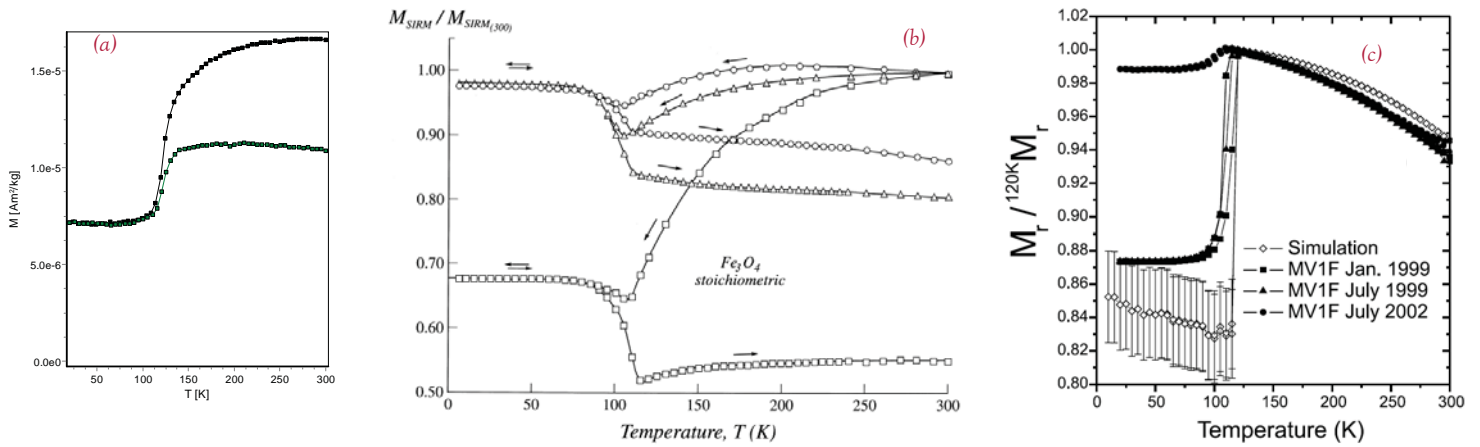


Fig 3. LTD of RTSIRM for samples containing SD magnetites. (a) silicate crystals from the Bushveld Complex with exsolved magnetite (Josh Feinberg, unpublished data). ‘First memory’  $\approx 50\%$ ; ‘second memory’  $\approx 75\%$ ;  $\Delta_{vj} \approx 0$ . (b) synthetic submicron nearly-stoichiometric magnetite (circles, triangles and squares indicate respective sizes of 37, 100 and 220 nm; magnetites were grown from solution and have low stress and very little shape anisotropy). From Özdemir et al 2002. ‘First memory’ ranges from 68% to 98%; ‘second memory’ from 55% to 85%; both decrease with increasing grain size;  $\Delta_{vj}$  is larger on rewarming than on cooling. (c) samples of magnetotactic bacteria containing chains of SD magnetite (squares and triangles show measurements on freshly-cultured samples; circles show measurements after oxidation due to storage in air for three years). For the stoichiometric samples ‘first memory’  $\approx 94\%$ ; ‘second memory’  $\approx 100\%$  as the curves are almost perfectly reversible; from Carter-Stiglitz et al (2004).

orientation of magnetization, regardless of grain shape. It has been commonly thought that in the absence of an applied field or magnetostatic interactions, one of the cube edge orientations is chosen at random to become the monoclinic c-axis. Rotation of the individual particle moments into c-axis alignment causes a loss of room temperature remanence, as some of the magnetization is effectively randomized (e.g. Fig. 3a,b,c). If there is no systematic relationship between crystallography and grain shape, the change from one uniaxial anisotropy to another results in a decrease of up to 50% in the net remanence of a population of grains.

When the sample is reheated, the moments might be expected to rotate reversibly back to their original direction with little to no loss of the original remanence. This appears to be true for some samples dominated by SD shape anisotropy at  $T > T_K$  (e.g., Fig 3c), because there are only two easy axes to choose from on re-heating; the shortest rotation path will almost always be a return to the starting room-temperature direction. Perfect reversibility is rarely seen in nature, however, and for particles with small degrees of elongation ( $<1.15$ ) it has been argued (Muxworthy and Williams, 2006) that cubic anisotropy indeed plays a role at  $T > T_v$ : the moment will find some orientation that is a local minimum in the sum of magnetocrystalline anisotropy energy (3 easy axes below  $T_K$  and 4 above) and magnetostatic (shape anisotropy) energy (2 or more easy directions). Not all of the individual particle moments return to their original orientations, and therefore there is incomplete recovery of remanence. This was demonstrated experimentally as early as 1978 when Levi and Merrill (1978) measured TRM remanence before and after cycling to  $T < 130\text{K}$ . They found that remanence recovery was greater in acicular grains than in equant grains. Likewise, interparticle magnetostatic interactions have been modeled to have a similar effect (Muxworthy and Williams, 2006), and can additionally reproduce intriguing features such as an additional decrease in  $M$  (rather than recovery) on warming back through  $T_v$  (Fig

3b).

#### Multi-domain, low- $T$ cycling of RTSIRM

In contrast to the SD case, remanence within the domains of larger particles almost always lies along one of the cubic  $\langle 111 \rangle$  axes for  $T > T_K$ . On cooling, the magnetocrystalline anisotropy energy decreases and domains reorganize irreversibly even at temperatures well above  $T_K$  (Halgedahl & Jarrard 95; Dunlop 2003; Liu and Yu 2004). For true MD populations (20  $\mu\text{m}$  and larger), 80% or more of RTSIRM can be erased by cooling to temperatures approaching but not reaching  $T_K$ . On cooling through the isotropic point, the cubic magnetocrystalline easy axes change to  $\langle 100 \rangle$ . Instead of coherent rotation of magnetization as in the SD case, the room-temperature domain configurations become unstable and walls may begin to move or dissolve, while new walls are nucleated. However in the temperature interval between  $T_K$  and  $T_v$ , the magnetocrystalline anisotropy remains weak, and only rarely is there a direct visible indication of  $T_K$  in remanence cooling curves (Fig 4; Özdemir & Dunlop, 1999). Similarly, Kasama et al (2012) saw no significant changes in domain arrangements in images obtained by Lorentz microscopy above and below  $T_K$ .

It is at  $T_v$ , with a tenfold increase in magnetocrystalline anisotropy and a change from cubic to monoclinic symmetry, that domain reorganization is most forcefully driven. MD grains are large enough for the monoclinic phase to nucleate and begin growing in multiple regions independently of one another, with c-axis selection determined (in zero applied field) either randomly, or by local stress fields, or by inheritance of the cubic-phase domain-moment orientations. Kasama et al (2012) observed strong similarities in the domain configurations above  $T_v$  and below it after cooling in zero field, indicating some control of the monoclinic c-axis selection by the pre-existing local magnetization (as suggested by Bickford



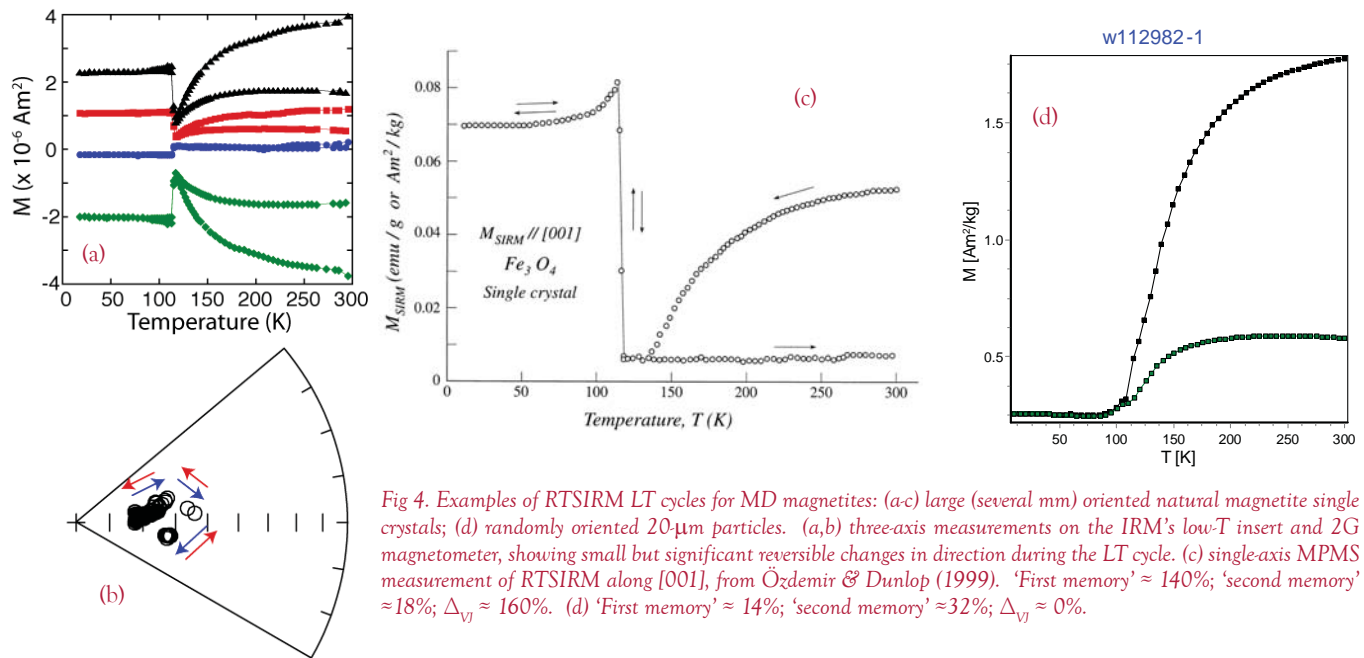


Fig 4. Examples of RTSIRM LT cycles for MD magnetites: (a-c) large (several mm) oriented natural magnetite single crystals; (d) randomly oriented 20- $\mu\text{m}$  particles. (a,b) three-axis measurements on the IRM's low-T insert and 2G magnetometer, showing small but significant reversible changes in direction during the LT cycle. (c) single-axis MPMS measurement of RTSIRM along [001], from Özdemir & Dunlop (1999). 'First memory'  $\approx 140\%$ ; 'second memory'  $\approx 18\%$ ;  $\Delta_{vj} \approx 160\%$ . (d) 'First memory'  $\approx 14\%$ ; 'second memory'  $\approx 32\%$ ;  $\Delta_{vj} \approx 0\%$ .

in 1953). For samples containing large MD grains, and especially for single crystals, the remanence often “jumps” on cooling through  $T_v$  in zero field (Özdemir & Dunlop, 1999; Muxworthy et al., 2003; see also *IRMQ v19 n4*); the magnitude of the increase depends on the nature of the initial remanence as well as on particle size. Changes in remanent magnetization on cooling and rewarming below  $T_v$  are almost always slight and perfectly reversible.

As the sample is warmed back through  $T_v$ , the strong monoclinic anisotropy abruptly vanishes, as does the monoclinic twinning, and domain walls will again unpin and reform. The c axis of each monoclinic twin domain becomes one of the cubic  $\langle 100 \rangle$  easy axes, so there is no moment rotation required by the change in magnetocrystalline anisotropy, but its diminished strength shifts the balance towards shape and magnetoelastic anisotropies. It is interesting that any “jump” in remanence associated with  $T_v$  is generally almost perfectly reversible, i.e.,  $M_5 \sim M_2$  (Fig 4).

On continued warming through  $T_K$ , the K1 anisotropy constant again changes sign and the cubic body diagonals again become the magnetocrystalline easy axes. There is no reason for the domains to reform precisely in the original room-T configuration, but Kasama et al (2012) have observed significant similarities in the patterns at 143 K before and after zero-field cycling through  $T_v$ . Nevertheless, in MD magnetites there is typically little or no recovery of the remanence lost on cooling. The “memory ratio” ( $M_6/M_1$ ) decreases systematically with increasing grain size from SD through MD grain sizes (Merrill, 1970; Levi and Merrill, 1978; Heider et al., 1992). Halgedahl and Jarrard (1995) found a nearly linear relationship between the memory ratio and the logarithm of grain size and, therefore, the number of domains.

It has frequently been suggested that this memory ratio can therefore be used to estimate grain size, but such an interpretation is predicated on the assumption of stoichiometric magnetite. This cannot be assumed in

many natural samples which will frequently have at least a small degree of cation deficiency or cation-substitution, both of which can have a strong effect on the memory ratio. Moreover the memory ratio in natural materials with mixed mineralogy depends on the relative proportions of phases that are invulnerable to LTD, and so granulometric estimation is inseparable from the more general unmixing problem. Additionally, as noted above, in the SD/PSD grain size region, acicular magnetite is found to have a higher memory ratio than comparably-sized equant grains.

A final complication in the interpretation of MD LTD memory lies in crystal defects and associated stress fields, which may serve as barriers to domain wall motion or as sites for wall re-nucleation, and which may thereby help to transfer magnetically-stored information back and forth between the cubic and monoclinic phases (Kobayashi and Fuller, 1968; Hodych, 1991; Heider et al 1992; Hodych et al., 1998; Kasama et al., 2012). Thus, the memory is a function of not only grain size but also stoichiometry and defects/stresses, which we will return to in future articles. Because LTD memory depends on these factors, it is not surprising that it also depends on the nature of the initial remanence.

### LTD of Different Remanences; Coercivity and Unblocking-temperature Spectra after LTD

Is it possible to isolate primary thermoremanence by selectively demagnetizing viscous and other overprints using LTD? Can the portion of NRM carried by MD grains be selectively removed by low-temperature treatments, leaving an ideal SD-carried remanence for paleointensity or paleothermometry studies? The answer to these is an unequivocal “Yes, to a certain extent, but...”

Interest in the use of LTD to isolate the primary TRM carried by SD grains has of course been particularly strong in connection with paleointensity, where only

such ideal records are amenable to techniques such as the Thellier-Thellier method, which depend on the laws of pTRM additivity, independence and reciprocity. Many studies have shown that the remanent “memory” of TRM and pTRM (and their anhysteretic analogues) after LTD have more SD-like properties than the untreated thermoremanences, including closer adherence to the virtuous properties of additivity, independence and reciprocity, but LTD pretreatments still commonly fail to yield reliable paleointensities from samples with MD magnetite (Dunlop & Argyle, 1991; Heider et al., 1992; McClelland et al., 1996; Shcherbakova et al., 1996; Muxworthy, 2000; Muxworthy & McClelland 2000a,b; Yu et al 2003; Yamamoto et al 2003; Borradaile et al., 2004; Dunlop et al., 2005).

### Multicycle LTD

Erasure of magnetic information by a single cycle through  $T_V$  is imperfect; some fraction is remembered in spite of the randomness inherent in phase nucleation, c-axis selection, domain nucleation, etc. Liu and Yu (2004) found experimentally that remanence decays exponentially with the number of LTD cycles, to a nonzero level that depends on the temperature endpoints of the LTD cycles, as well as on all the usual other factors like particle size and type of remanence. The first cycle is responsible for more than 75% of the total remanence loss caused by 10 or more LTD cycles, when the cycles cross  $T_K$  but not  $T_V$ . For lower-temperature cycles that cross  $T_V$  but not  $T_K$ , the first cycle accounts for about half the total demagnetization accomplished by 10 or more cycles. Liu and Yu (2004) modeled the process using a “Boltzman-analog” approach involving both domain reorganization above  $T_K$  and easy-axis reorientation through  $T_K$  and  $T_V$ .

### Experimental Artifacts

No article on the fine details of low-temperature magnetic behavior would be complete without some mention of various nefarious experimental artifacts that torment and bedevil us on a regular basis, by mimicking and/or distorting the signatures of the Verwey transition and other significant features. In the context of LTD, the most bothersome issue involves spurious abrupt decreases in remanence while cooling in zero field, due to reorientation of particles in powder samples or other similar effects. Typically these appear as discontinuous steps superposed on a smoothly changing signal (e.g., Fig 5). With surprising frequency the anomalous drops occur within the temperature ranges where we look for the Morin transition (hematite), the Verwey transition, the Besnus transition (pyrrhotite), or different mineral isotropic points, and it can be difficult to evaluate their significance. Repeat runs commonly show that they are not reproducible in detail, or often even in gross form; repacking the sample more tightly to immobilize the particles more completely in many cases eliminates the artifacts. It is conceivable that particle reorientation is itself related in some way to the

phase transitions, and the abrupt drops may still in some cases be a guide to the presence of transitions. However, in order to obtain unambiguously interpretable results we recommend, when practical, working with solid rock chips/slices, or mixing granular materials with a compressible binder to reduce or eliminate the occurrence of such gremlins.

### Closing Remarks

In the nearly fifty years that have passed since the first work on LTD, it has continued to draw interest and stimulate research, even if it has not quite become a staple of paleomagnetism in the same way that thermal and alternating-field demagnetization methods are. Stepwise LTD (e.g. Dunlop 2003) and full 3-axis magnetization measurements during low-T cycles (Bowles et al, 2010; Smirnov & Tarduno, 2011) promise to improve our understanding of natural and artificial remanences carried by magnetite, and direct imaging of cubic and monoclinic crystallographic and magnetic microstructure (Kasama et al., 2012) will continue to clarify their interdependent changes across  $T_V$ , deepening our understanding of transition-related phenomena as well as of the stabilizing mechanisms of room-temperature remanence. Behavior of room-T remanence during LTD provides information on the mineralogy, grain sizes, stoichiometry and anisotropy of the carriers, and for these reasons magnetization measurements across low-temperature transitions have become, and will continue to be, essential for studying the sources (minerals and mechanisms) of stable remanence in natural materials.

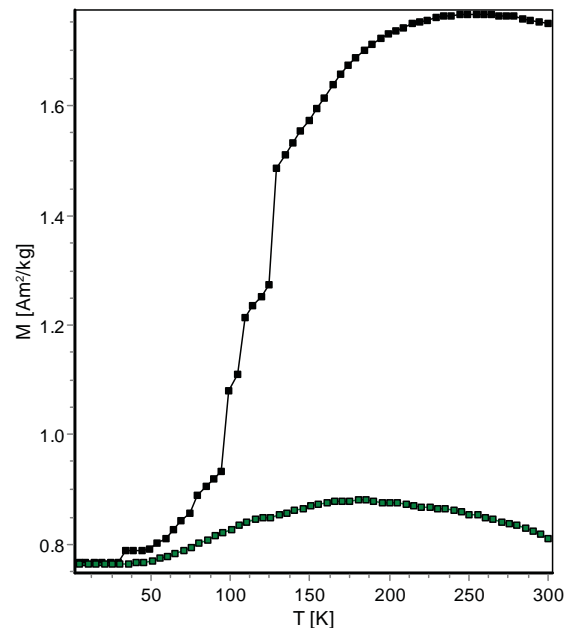


Fig. 5. Frustrating inexplicable mystery discontinuities commonly found in cooling curves on powder samples, often tantalizingly close to significant transition temperatures. Repacking and remeasuring sometimes eliminates these.

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